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# ***JPRS Report***

# **Science & Technology**

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***Japan***

4TH FUNCTIONALLY GRADIENT MATERIALS SYMPOSIUM

SCIENCE & TECHNOLOGY  
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4TH FUNCTIONALLY GRADIENT MATERIALS SYMPOSIUM

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## Effect of Compositional Profile on the Thermal Stress in FGM

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### Abstract

Heat and stress analyses are carried out by means of FEM to obtain the optimized compositional profile of FGM. Under the condition of high temperature difference, thermal stress generated in the FGM decreases with the increase of ceramic composition. In case of the rigid cooling surface, the stress component in the thickness direction becomes critical at the fixed surface. On the other hand, under high heat flux condition, the increase of metallic composition in FGM introduces the decrease of thermal stress. The verification of the results of FEM analysis will be conducted by the simulation experiments.

For the development of suitable design system of FGM, it is necessary to prepare the material data base and the detail boundary condition for analysis.

## Micromechanical Approach to the Elastic-Plastic Analysis of Thermal Stresses in a Ceramic-Metal FGM Plate

Kenji Wakashima and Hideaki Tsukamoto

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### Abstract

A "piecewise linear" elastic-plastic formulation is given for numerical analysis of transient thermal stresses in a ceramic-metal Functional Gradient Material (FGM) plate with in-plane isotropy. It is assumed that the plate is thin enough for the plane stress condition to apply and the temperature varies only through the thickness of the plate. Using an established "mean-field" theory of the micromechanics of multiphase systems, a closed-form expression for the effective elastoplastic compliance in balanced biaxial tension is derived with particular reference to the microstructure made up of randomly dispersed spherical particles.

## Elastic-Plastic Thermal Stress Analysis for Optimum Design of FGM

Yoshio Arai, Saitama University, Faculty of Engng., Dept. Mechanical Engng.

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Dept. Mechanical Engng. Sci.

### Abstract

Equivalent inclusion method was applied to FGM to evaluate the elastic constants and thermal expansion coefficients. Thermal and stress analysis of FGM were performed by 2-D FEM. Sintered residual stress, thermal stress at thermal loading and total residual stress after cooling were studied. The example for the optimum design for FGM was presented.

## Ultrasonic Testing of Thermal Shock Damage on Plasma Sprayed FGM Coating

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Nondestructive Evaluation/Strength Evaluation Section

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### Abstract

This paper describes the results on immersion c-scope ultrasonic testing of thermal shock damaged FGM coatings. Roughness of sprayed coating surface makes it difficult to detect surface crack produced by CO<sub>2</sub> laser irradiation. We investigate several measuring technique and make clear that transmission echo of coating layer obtained by back surface echo measurement contains much information on thermal shock damage.

# Measurement of Young's Modulus and Poisson's Ratio for Functionally Gradient Materials by Using Line-focus-beam Acoustic Microscope

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## Abstract

Purpose of this paper is to develop the non-destructive elastic constant measurement procedure of Functionally Gradient Materials by using line-focus-beam acoustic microscope. Effective velocity measurement area of acoustic microscope is narrow enough for the uniform materials, however insufficient for the FGM which has nonuniform narrow layers. Then, in order to narrow down the measurement area, new analytical procedures are added in  $V(z)$  curve analysis method for velocity measurement. Accuracy of the elastic constant obtained by the velocity measurements based on elastic theory are investigated comparing to the that of 25 non FGM specimen. As the result, narrow area analysis procedures are so effective for elastic constant estimation of FGM, and accuracy of estimated elastic constant considered highly enough for industrial application. Elastic constant dependence on production condition of FGM is also investigated.

## Nondestructive Evaluation of Functionally Gradient Materials Using Ultrasound (1)

Kazuo Fujishima, Hiroshi Yamamoto, Yasuo Hayakawa, Toshio Nonaka  
Technical Research Lab., Hitachi Construction Machinery Co., Ltd

## Abstract

Ultrasonic measurements at high frequency are expected as nondestructive inspection methods for ceramics, composites and functionally gradient materials. Measurements are divided to two main methods in industry. One is suitable for detecting fine flaws in the opaque materials, and another is available for characterization of materials. In this report, these two methods are used to evaluate ceramics received heat-shock by laser irradiation on the surface. The shapes of cracks opening on the surface are measured by ultrasonic testing with impulse wave. It is observed that the cracks, which appear on the surface, propagate slantingly inside and then stop their growth. Material characterizations are measured by ultrasonic microscope with tone burst wave. It is found that the surface wave velocity at damaged area is lower than that of at less-damaged areas, and that the Young's modulus at damaged area is lower than that of at less-damaged areas. Ultrasonic microscope have possibility to evaluate the damage on the surface.



## Nondestructive Evaluation of Functionally Gradient Materials Using Ultrasound (2)

Hiroyuki Nishimori, Yasuo Hayakawa and Toshio Nonaka  
Technical Research Lab., Hitachi Construction Machinery Co., Ltd

### Abstract

This paper presents some results of ultrasonic inspection for Functionally Gradient Materials (FGM) which consist of multi-layer with varying mixture rate of  $ZrO_2$  (with 8%  $Y_2O_3$ ) and Ni-based alloy (NiCrAlY). FGM are received heat-shocked by laser irradiation on the surface. The flaw detection and material characterizations are measured by Ultrasonic Testing with impulse wave and Scanning Acoustic Microscope (SAM) with tone burst wave. In a polished surface sample, it is found that the surface wave velocity differs at mixture rate, and that at damaged areas is higher than that at less-damaged areas. Micro cracks which are not found by the optical microscope are observed. In a sample with rough surface, some inside cracks are detected.

## Fabrication of sintered SiC-AlN/Mo Functionally Gradient Materials

Masahiro Tanaka, Akira Kawasaki, Ryuzo Watanabe  
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### Abstract

The Powder metallurgical fabrication of the functionally gradient material for thermal barrier use of a specific material combination of SiC/AlN/Mo is described. Results of thermomechanical tests for non-FGM have shown that the samples HIP'ed at 2123K for 2h from mixture of SiC and AlN powders have a sufficient thermal barrier property with high fracture strength even at elevated temperature than monolithic SiC and AlN. The designing of the compositional distribution was made on the basis of the calculation of the overall heat transfer in a temperature difference of 1000K.

According to the optimum compositional distribution, the sintered compact of the functionally gradient material of SiC/AlN/Mo was successfully fabricated.

## Fabrication of Sintered Functionally Gradient Material by Dipping Process

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Ryuzo Watanabe Faculty of Engineering, Tohoku University

### Abstract

In the synthesis of sintered FGM, the most important problem is how to stack the mixed powders as low materials according to the designed composition profile.

This study aims to develop the dipping process which makes it easy to stack the powders on substrates of complicated shapes. In this process, the powders are deposited on the substrate which is dipped into the ethanol slurry containing the mixed powders with desired composition. Successively, this substrate is dipped again into the slurry containing another mixed powders.

These dipping processes are repeated to realize the depositions with the desired gradient composition. Deposited product is dried, CIP ed, encapsulated and HIP ed.

## Development of Large-sized FGM by Thin Sheet Lamination Method

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### Abstract

Small-sized FGM (30mm  $\phi$ ) of zirconia and nickel has been developed by the thin sheet lamination method. To evaluate the thermo-mechanical properties of FGM, it is indispensable to develop the FGM actively-cooled panel. So a large-sized FGM (60 $\times$ 60mm) was manufactured for trial. The problem for enlargement is warp-control. The warping which occurs in as-sintered FGM was measured and reform-press was used to flatten the FGM. And the reform-press was certified as effective. Thus, it is possible to braze the FGM and the panel.

## Packing of Alumina-Nickel Mixture With Continuous Composition Profile

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The packing technique which can smoothly vary the composition of formed material is essential to develop a so-called functionally gradient material. In this paper, the packing method based on filtration operation is investigated. Continuous change of the composition of filtered slurry by mixing two slurries gives the filter cake with composition profile. The profile corresponds to the mixing process and can be easily determined from the mass balance for the filtration process. Experiments for alumina-nickel composites support the availability of the present technique. F test was used to check the mixing degree of the two components. Using Buslik's complete mixing theory, it was safely concluded that the complete mixing was attained at any layer of the composite.

## Al-AlN FGM Made with an Ultrafine Particle of Al-AlN Mixture, and Its Usage

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### Abstract

The ultrafine particle of Al-AlN mixture was prepared by means of the arc-plasma processing that a vapourized and fumed substance in nitrogen-hydrogen-argon atmosphere was collected into a bag filter. The ultrafine particle was partially Al metal and partially AlN. Al metal in the ultrafine particle reacts with nitrogen into AlN at 923 K. A commercial powder of Al metal, however, does not react with gaseous nitrogen, but it is easily sintered to a compact block at 923 K. The mixture of the ultrafine particle and the commercial Al powder, whose contents were gradually changed, was pressed in a cylindrical steel die under pressure of 20 MPa. The green compact which was prepared in this manner was heated at 923 K in nitrogen flow. The FGM block of the system Al-AlN prepared by this method was characterized some properties and was considered some applications for joinings.

## Development of Functionally Gradient Materials of Foam Glass Composite

Shinichiro Kato, Kazushi Sato, Hideaki Takahashi

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Faculty of Engineering, Tohoku University

### Abstract

This paper presents some results of transient thermal stress analyses of functionally gradient foam glass subjected to rapid heating. The objective of this study is to examine the effect of changing the density's distribution profiles in FGMs on the steady thermal stresses. Finite element analyses are conducted on FGMs which possess different gradient of density in the foam glass. A two-dimensional, axisymmetric model is used for the numerical computations, where the surface is subjected to a heat flux and the back temperature is kept at 20°C. Additionally, the foam glass sample and the layered foam glass by Wollastonite were evaluated the thermal shock test/AE technique. The experimental results indicates an improvement of the thermal shock resistance by Wollastonite.

## SiC/C Functionally Gradient Materials Prepared by Chemical Vapor Deposition/Infiltration

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Central Technical Research Laboratory  
Nippon Oil Company, LTD

### Abstract

SiC/C Functionally Gradient Materials(FGM) were prepared by Chemical Vapor deposition/Infiltration(CVD/CVI).  $\text{SiCl}_4$ ,  $\text{CH}_4$  and  $\text{H}_2$  were used as source gases. The microstructures of non-FGM and FGM were studied in detail. The non-FGM with carbon content 100 mol% showed various thermal properties with the preferred orientation of carbon. The pore size distribution of non-FGM with various SiC/C ratios were characterized by the mercury porosimeter. SEM and EPMA analysis were also carried out to reveal the microstructures. Carbon/Carbon composites coated with SiC/C-graded coating were tested in the rocket combustor environment and the high temperature different field. FGM coated Carbon/Carbon composites showed good results in comparison with conventional coated Carbon/Carbon composites.

## Fabrication of Functionally Gradient Material of TiC-SiC system by CVD.

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Sumitomo Electric Industries, Ltd.

### Abstract

Composite materials of the TiC-SiC system were fabricated using the CVD process and their thermal and mechanical properties were measured. The relationship between the properties and microstructures of the composites and the possibility of developing into Functionally Gradient Materials (FGM) were then investigated. The CVD conditions used were,  $\text{TiCl}_4$ - $\text{SiCl}_4$ - $\text{CH}_4$ - $\text{H}_2$  system, deposition temperature of  $1350^\circ\text{C}$  and total gas pressure of 60 Torr. The TiC-SiC composites were fully dense with columnar structure perpendicular to the substrate plane, and their thermal expansion coefficients and Young's Modulus respectively changed from  $4.5 \times 10^{-6} \text{K}^{-1}$ , 220GPa for SiC to  $7.4 \times 10^{-6} \text{K}^{-1}$ , 467GPa for TiC, depending on the composition. Based on thermal stress analysis calculated from the data obtained by subsequent experiments, compositionally gradient TiC-SiC layer was formed as an intermediate layer in oxidation resistant SiC coating on carbon-fiber reinforced TiC composites with a thermal expansion coefficient larger than that of SiC. Consequently, a coated layer with non-peeling was obtained. It was demonstrated that the residual stress in the surface layer of SiC was reduced by introducing the compositionally gradient layer of TiC-SiC system as an intermediate layer.

## Development of SUS316L/Cu Gradient Material by Sintering and Infiltration Technique

Masashi Takahashi, Yoshiyasu Itoh, Matsuo Miyazaki,  
Hideo Kashiwaya and Shigeru Adachi  
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### Abstract

High strength at elevated temperature, sodium (Na) corrosion resistance and radiation toughness are necessary for fast breeder reactor (FBR) components. The application of functionally gradient materials for them is one of the superior method to stand against the several conditions described above. In particular, it is thought that a SUS316L/Cu gradient material is effective to reduce the thermal stress which generates for the temperature gradient. The spraying and infiltration technique, which is developed to fabricate the SUS316L/Cu gradient material, is shown in this paper. The technique consists of two steps. The first step is to fabricate sprayed SUS316L layers with gradient and the second step is to infiltrate a molten Cu in the gradient pores. In this study, it is confirmed that SUS316L/Cu gradient material can be fabricated by the spraying and infiltration technique, and reactions between SUS316L and Cu can't be observed in HIP condition of 1100 °C, 196MPa, 30 minutes.

### Al-Mullite-SiC Fiber FGM by Low Pressure Plasma Spray

Toshio Sakakibara, Eiji Tanikawa, Masakazu Shimanuki  
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### Abstract

The development of basic technique for Functionally Gradient Materials (FGM) reinforced by SiC fiber using Low Pressure Plasma Spray was carried out. Al-Mullite mixed powders were sprayed on the winding interval-controlled-SiC fibers. The preforms were fabricated by the repeat of spraying layer by layer, while the mixing ratio of the powders and winding intervals were changed gradually in the thickness direction. After spraying, the preforms were hot-pressed in order to compact the matrix, especially in the Al rich layers. The trial products made by this process were examined by the 4 point flexure test. Consequently, it was found that fiber reinforced gradient materials had 4 times higher strength than non-reinforced gradient materials.

In this report, structural, thermal and mechanical properties of Al-Mullite combination materials were described, in addition to the results of the fiber reinforced gradient materials.

## Developing a Fabrication Process for FGM

By Using a Low Pressure Plasma Spraying

Saburo Kitaguchi, Hideki Hamatani, Nobuyuki Shimada,

Yasutomo Ichiyama and Tohru Saito

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### Abstract

This study was aimed at developing a fabrication process for FGM by using a Low Pressure Plasma Spraying (LPPS). Experiment of YSZ-NiCr FGM spraying showed that the vacuum condition and the initial powder size was important parameter for depositing dense coatings. Under optimum conditions, the density of coating exceeded 95% theoretical. Maximum temperature and temperature distribution in substrate during pre-heating and spraying, had a large effect on reducing a decomposition of substrate. And thermal fatigue properties and thermal stress relaxation were carried out to investigate by cyclic high temperature difference filed test. The results of this test cleared the optimum design about profile and density for those properties. Successive sprayed position of FGM had proved that LPPS must be a strong candidate to open new aspects of production process for large size FGM.

## Production of a Functionally Gradient Material by SHS Process and the Heat Shield Property

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### Abstract

Total production process of Functionally Gradient Material by SHS process (SHS-FGM) have been described.  $TiB_2$ -Cu-FGM have a highly heat conductivity. To gain heat shield effect in the  $TiB_2$ -Cu-FGM,  $ZrO_2$  was added as a rare material because of the lower heat conductivity of 1/30 than  $TiB_2$ .  $TiB_2$ -Cu-FGM and  $TiB_2$ -Cu- $ZrO_2$ -FGM have been tested to measure the temperature drop from the surface and the back side of the FGM. Although the result is clear that there is more heat shield effect than  $TiB_2$ -Cu-FGM, we have to develop more effective by considering the compositional profile and the thickness of FGM layer, etc.



# Effect of Hydroxyapatite Coating on Porous Metal in Order to Have Functional Gradient Effects on the Interface Between Bone and Artificial Bone and Joint

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- 2) Sumitomo Chemical Co.
- 3) Sumitomo Heavy Industries Co.
- 4) Osaka Prefectural Industrial Engineering Institute

## Abstract

Artificial tibiae with two stems coated with two layers of titanium alloy beads (Ti-6Al-4V, bead diameter of 350-400  $\mu$ m) were further coated with hydroxyapatite (HAp). The six beagles were divided, four in the HAp-coated group and two in the HAp-uncoated group. The materials used for 6-month and 3-month evaluations were implanted in right and left tibia respectively. Since the amount of bone ingrowth into porous spaces was larger in a stem having a porous coating when implanted in the cortical bone than in the cancellous bone, the fixability of the stem was more stable and firmer in the cortical bone, and the incidence of stem loosening was also lower in the cortical bone. When the porous coating was further coated with HAp, new bone tissue had earlier access and contact to the surface of beads in the superficial layer of the stem in contrast with the porous coating without HAp coating. Furthermore, the new bone tissue entered into porous spaces earlier and in larger amounts, could reach the deepest region, and bound to the beads. Thus the stem was able to bind directly to the bone within an early post-operative period. Since the state of bone ingrowth did not differ between 3 and 6 months after implantation, bone ingrowth would be attained within 3 months after implantation.

## Evaluation of Thermal Shock Resistance of SiC/C Functionally Gradient Material

Makoto Sasaki, Toshio Hirai

Institute for Materials Research, Tohoku University

## Abstract

A "Functionally Gradient Material, FGM", which has compositional gradient from one side to the other in the material, has been fabricated using CVD processing. Thermal shock resistance of the SiC/C FGM was evaluated by CO<sub>2</sub> laser. These results lead to the conclusion that the SiC/C FGM has high thermal shock resistance.



## Thermal Stability of FGM in Uniform and Gradient Temperature Fields

Yoshikazu Shinohara, Yoshio Imai, Susumu Ikeno, Ichiro Shiota

5th Lab. Physical Properties Research Division,

National Research Institute for Metals, STA

### Abstract

Fundamental researches were carried out to improve thermal stability of functionally gradient materials(FGMs). Specimens were FGMs and NFGMs(non-FGMs) of Ni-Cr-Al-Y/PSZ system fabricated by plasma spraying, Ni/PSZ by powder configuration process, C/SiC and TiC/SiC by CVD and Cu/TiB<sub>2</sub> by self propagating high temperature synthesis(SHS). Morphological and constitutional changes in uniform and gradient temperature fields were investigated. FGM structure is homogenized or separated to two phases at an elevated temperature. The following advice to fabricate FGMs with higher thermal stability were obtained.

- 1) It is very important to find the stabilizing system of FGM structure for its practical application.
- 2) Porosity of FGM should be decreased to restrain the volume change by sintering.
- 3) It is important to control the porosity and thickness of ceramics part to restrain crack initiation.
- 4) When material combination of FGM with the substrate is selected, it is necessary to mention the diffusion between the FGM and the substrate at an elevated temperature.

## A Study on Thermal Fatigue of Functionally Gradient Materials Under High Temperature Differences

Masaki Sasaki, Akinaga Onakawa,	Mamoru Takahashi,
Masayuki Niino, Masahiro Sato	National Aerospace Lab.
Kazunobu Aoyama, Satoshi Nagata,	Shinichiro Miyoto
	Mitsubishi Heavy Industries, Ltd.
Yoshinari Miyamoto	Osaka University
Nobuhiro Sata	Government Industrial Research
	Institute, Tohoku
Tohru Saito	Nippon Steel Corp.
Hideyuki Arikawa	Hitachi, Ltd.
Yukinori Kude	Nippon Oil Co., Ltd.

### Abstract

The thermal fatigue properties of some kinds of Functionally Gradient Materials (FGM) specimens,  $TiB_2/Cu$ , PSZ (Partial Stabilized  $ZrO_2$ ) /Cu, PSZ/Ni-Cr, SiC/ C/C and TiC/Ni-FGM, were characterized by means of the effective thermal conductivity,  $K_{eff}$ , under high heat flux conditions in the temperature different field.

It was observed that the thermal fatigue properties of  $TiB_2/Cu$  and PSZ/Cu were superior because the deterioration of normalized  $K_{eff}$  were small by 30 or more thermal cycles. In SiC/ C/C, the value of  $K_{eff}$  of FGM less decreased than that of Non-FGM (NFGM) as the number of thermal cycle increased. It was inferred that FGM prevented the peeling of surface SiC coating layer.

In TiC/Ni-FGM, four FGM specimens which had different compositional distribution were tested. It was experimentally confirmed that there was an optimum compositional distribution against thermal fatigue.

## High-Temperature Supersonic Gas Flow Heating Test of Cooled FGM Panel

Yoshio Wakamatsu, Toshihito Saito, Noboru Sakuranaka, Shuichi Ueda,

Tatsuo Kamagai, Kazuo Kusaka, Masahiro Takahashi, Katsuto Kisara,

Takayuki Sudo, Masayuki Niino : NAL-KRC

Masataka Yamamoto : NASDA .

Satoshi Nagata : MHI

### Abstract

An experimental test program is in progress to evaluate the structural safety degrees for the Functionally Gradient Materials (FGM) under the condition of high-temperature and supersonic air-flow. These condition is almost the same with that of the nose-cone and intake section of scramjet engine for the space-plane. Preliminally test was conducted to obtain additional data for examining possible merits of the experimental test. In this test of  $\text{NTO}/\text{N}_2\text{H}_4$  propellant system, it was found that the heat-flux for the nickel test sample was of the order of  $3\text{MW}/\text{m}^2$  at a section inclined by 30 degrees to the chamber axis. We are underway to operate the hot gas generation system using the gas condition of  $\text{H}_2/\text{O}_2/\text{N}_2$  in order to evaluate the FGM in November 1991.

## On the Fundamental Design for FGM and the Multi-Objective Optimization

Tohru HIRANO, Tomohiko YAMADA, Junichi TERAOKI

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Daikin Industries, Ltd.

### Abstract

In this paper, the fundamental design procedure for FGM is presented. At first, distribution functions are introduced, and estimation methods for the effective material properties of the intermediate compositions are explained. Then, thermal stress calculation methods for different processes and geometrical configurations are described. Finally, the multi-objective optimization problem is defined for the design process of FGM and solved by several methods including application of Fuzzy Set Theory.

Development of Two Dimensional FEM Program for an Analysis  
of Transient Thermal Stress in FGM

Junichi Teraki, Tohru Hirano  
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Abstract

In this paper, we present a result of an analysis of thermal stress in circular plates of Functionally Gradient Material(FGM). We develop two dimensional FEM program to analyse the transient heat conduction and the thermal stress in FGM. By means of this program, we simulate the thermal stress of Zirconia(PSZ) / Steel(SUS) FGM in high temperature gradient environment, reveal the transient effect and local heating effect.

Steady Thermal Stresses in a Plate of Functionally Gradient Material  
(Influence of Mechanical Boundary Conditions)

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Naotake NODA and Tomoaki TSUJI, Shizuoka University.

Abstract

A functionally gradient material which decreases thermal stresses has been developed for structural components and/or mechanical elements in fields such as nuclear, aircraft and space engineering. Steady state thermal stresses in a plate made of the functionally gradient material are discussed. The main theme of this subject is how to distribute the component of the material in the functionally gradient material to decrease thermal stress for different mechanical boundary conditions. In estimation of stress ratio which means the safety factor, the compressive strength of the material was considered too. We discussed the general method to decrease thermal stress in laminated plates made of two and three layers. The plate of an optimal functionally gradient material was determined to increase the safety factor.

## Fundamental studies on TiAl-Base Functionally Gradient Materials

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Shun-ichi Minakata, Hiroyasu Nishikawa, and Takao Yoshikawa  
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Yoshinari Miyamoto

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### Abstract

TiAl intermetallic compounds, owing to their low density and their outstanding high-temperature strength, are expected to be applicable to hypersonic transport(HST) aircraft. The aim of this study is to develop TiAl-based Functionally Gradient Materials(FGMs) with superior heat insulation and thermal stress relieving functions, in order to apply them to those components of an HST aircraft that would be subject to extremely large heat loads.

First, the properties required of heat-insulating FGMs were collated, and then, possible material systems that might satisfy the requirements were studied. As a result, it is concluded that the most promising material system would be a multiphase FGM of  $Al_2O_3$ -SiC-TiC-TiAl which has lower density, excellent thermal insulation property, thermodynamical stability, and oxidation resistance. Further more, the heat transfer properties and thermal stress properties of the multiphase FGMs were examined analytically, and a possible compositional distribution of the FGM was proposed.

### Study on Bonding of Functionally Gradient Material to Actively-cooled-Panel

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Masayuki Niino, Akinaga Kumakawa

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### Abstract

In the real condition tests Functionally Gradient Material(FGM) must be actively cooled to generate high temperature gradient in its depth direction. To assure cooling property between the test, bonding of FGM and actively-cooled-panel is very important. So thermal/structural analyses and fundamental experiment for C-SiC FGM on C/C composite brazed with actively-cooled-panel were performed. Two and/or three dimensional transient thermal/structural analyses were performed on the conditions of brazing and high-enthalpy gas exposure test. And optimization of brazing parameter were also investigated for the fabrication of actively-cooled-panel with FGM.

Fabrication Process of Actively-Cooled-Panel With FGM Fabricated by  
Thin Sheet Lamination Method

Chikara Fujiwara, Satoshi Nagata, Shinichiro Kiyotoh

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Akinaga Kumakawa, Masayuki Niino

Kakuda Research Center, National Aerospace Laboratory

Manabu Tamura, Tamako Hyakubu

Steel Research Center, NKK Corporation

Abstract

Fundamental Study on brazing of FGM fabricated by thin sheet lamination method to metal plate was carried out to develop the fabrication process of actively-cooled-panel with FGM. The FGM is designed to reduce thermal stress in service where the top surface is heated heavily and the bottom surface is cooled with coolant resulting in large temperature difference between both surfaces. The FGM is constituted of zirconia and nickel, and the composition is varied gradiently from the top surface to the bottom surface. Thus, a layer in the FGM is different in coefficient of thermal expansion from any other layer. Consequently, the FGM changes inherently its shape as its temperature varies uniformly such as in the case of brazing. Therefore, investigation was begun with the evaluation of the flatness difference of the FGM plate between at room temperature and at brazing temperature. Then, brazing parameters were optimized to establish the fabrication process of actively cooled panel with FGM.

## Development Plan of FGM Project 2nd Phase

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### Abstract

With a view to establishing the technology fundamental to creating FGMs for easing thermal stress in an environment with a maximum surface temperature of 2000K and a gap in temperature of 1000K, the project has succeeded in creating small sample pieces, 1~10mm thick and 30mm in diameter. This was a major success during its first phase, FY1987~1989. The second phase, FY1990~1991, has seen as its primary goal the production of 300mm square shells.

The latest achievements are given below.

### Studies on Microstructures and Fracture Mechanism of FGM Prepared by Low Pressure Plasma Spraying

Hideki Hamatani, Nobuyuki Shiida, Yasutomo Ichiyama,

Saburo Kitaguchi and Tohru Saito

Joining Steel Research Laboratories, Nippon Steel Corporation

### Abstract

Studies on microstructures and fracture mechanism of FGM, consisted of YSZ and NiCr, prepared by LPPS were carried out. MSP test was performed to measure the fracture stress. Cyclic high temperature difference field test was used to investigate thermal fatigue properties. The microstructure of NiCr showed a columnar structure and its growth was perpendicular to the interface. On the other hand, in case NiCr was surrounded by YSZ, its microstructure was found to show an equiaxed structure. Initial particle size had a large effect on density, grain size and fracture stress. The size for obtaining maximum fracture stress was not coincident with that for the maximum density of coatings. Through the results of high temperature difference field test, an optimum design to reduce thermal stress has been proposed.

# Thermal Evaluation Test of Functionally Gradient Materials

Made by Low Pressure Plasma Spray

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Joining, Steel Research Laboratories,

Nippon Steel Corporation

Ryuzo Watanabe, Akira Kawasaki, Atsushi Hibino

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## Abstract:

For the better understanding of the fracture process of Functionally Gradient Materials (FGM) in the temperature difference field, burner heating test, using hydrogen and oxygen, was carried out. Plasma-sprayed FGM, consisting of zirconia and nickel alloy were prepared in this testing. Vertical cracks were observed in a ceramics (top) layer, and terminated mainly at metal layers in a metal/ceramics mixing area. This is thought to suggest the advantage of FGM for its vertical crack arrestability. Horizontal cracks, on the other hand, were found in a ceramics (top) layer and propagated between vertical cracks. In addition, some horizontal cracks which did not follow the vertical cracks have also been found through the observation.

## Property Improvement of gradient coatings Formed by Plasma Twin Torch Spraying Process

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and Seiji Kuroda

4th Lab. Advanced Materials Processing Div., National Research Institute for Metals S'A

For the purpose of property improvement of gradient coatings formed by plasma twin torch spraying process, heat treatment was tried to the deposited coatings. Coatings were formed by deposition of Ni-Cr-Al-Y alloy (Ni base alloy) and  $Y_2O_3$  (8%) stabilized  $ZrO_2$  ceramics (YSZ) using plasma twin torch spraying process, and heat treatment was carried out at conditions of 1473K, 144h under a pressure of  $1.33 \times 10^{-2}$  Pa.

In the results of this experiment, it was recognized that heat treatment for Ni base alloy and YSZ coatings was effective to improve the denseness, uniformity and cohesive strength of the coatings.



# Fabrication of Plasma Sprayed Intermetallic Matrix Composite Coatings as a Light Weight Heat Resistant Material

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## Abstract

Both Ti-Al and Mo-Si powders, which were highly mixed and bonded of each raw metal materials by the mechanical alloying, were introduced to rf plasma spraying. DC plasma spraying was also conducted under the equivalent experimental conditions to compare with the rf plasma spraying results. To fabricate the intermetallic matrix composite coatings, Ti-Al/Si<sub>3</sub>N<sub>4</sub> MA pre-composite powders were made and plasma sprayed. Whereas the complete Ti<sub>3</sub>Al intermetallic coatings could not be obtained by dc plasma spraying, Ti<sub>3</sub>Al intermetallic coating was fabricated by rf plasma spraying without after heat treatments. Intermetallic matrix composite coatings were also made in the similar spray processes of Ti-Al/Si<sub>3</sub>N<sub>4</sub> system. The phase obtained by spraying of Mo-Si MA powder was fairly complicated. More detailed observation was required in this system.

## Microstructural Dependence of Mechanical Properties for Sintered Functionally Gradient Material

Shunsuke Yamakawa, Akira Kawasaki, Ryuzou Watanabe

Faculty of Engineering ,Tohoku University

## Abstract

This paper reports an evaluation of the mechanical properties of metal/ceramic composite in relation to microstructural features. A zirconia /stainless steel mixed composite having definite metal/ceramic ratios were fabricated by powder metallurgical method. A variation of flexural strength has been found to depend on the composition and microstructure. The AE analysis for the fracture behaviors during crack propagation during loading in the indentation fracture test along with microstructural investigation, was made. It has been found that fracture behaviors are strongly dependent on the continuity and morphology of the metallic phase; that means that the skeletal to dispersive structural transition and the cluster formation dominate the fracture behaviors.

## Joinings of Metal or Alloy to Magnesia Using Functionally Gradient Materials

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### Abstract

In order to obtain the joints of nickel and magnesia, a functionally gradient material of Ni-NiO was used for joining fillers. The joints of permalloy and magnesia were also done with the same fillers. The joints of cobalt and magnesia were accomplished using a functionally gradient material of Co-CoO. Furthermore, in order to obtain the joints of nickel and magnesia, I tried to use a functionally gradient material of Ni-MgO. Such a joint has no flat joining interface which are weak point against fractures. Thus, bending strength of the joints increased than that of the direct joints of metal or alloy and magnesia. It seems that the FGM block filler to joinings is useful to reduce residual thermal stress.

The FGM block was made by the process of powder metallurgy. The several layers consist of metallic powder and the mixed powder of metal and metal oxide, whose composition gradually changed, were compacted in a steel die under pressure. The compacts were sintered at 1573 K for several hours in air.

## Study on the Cooling Structure to FGM

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### Abstract

In the thermal fatigue evaluation test for FGM at high temperature difference, the ceramics rich side of FGM is heated and the opposite side is cooled by coolant through the grooves in the cooling structure that is bonded to the metal rich side of FGM. In case of bonding the cooling structure to the FGM, it is necessary to avoid the deformation of the FGM by heating process, so the Electro Forming Process is a suitable method, because it is free from heating process. The Ni-Electro Forming Process of the small panel for the thermal fatigue test of FGM was investigated.

**Temperature Response and Evaluation of Thermophysical Properties  
of Functionally Gradient Materials  
in Transient Measuring Methods**

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**ABSTRACT**

Wide attention has been given to multi-layered materials as electronic materials and materials resistant to wear, corrosion, and heat; they are expected to improve the specific nature of the conventional materials with homogeneity in composition, structure and texture. Among these advanced materials, functionally gradient materials (FGM), which are composed of different components such as ceramics and metals with continuous profiles in composition, have attracted special interest as advanced heat-shielding structural materials in future space applications.

The objectives and contributions of the present study are the following. First, an analytical solution of the temperature rise in the multi-layered material, which is subjected to the transient heating, is developed. This solution is easily extended to the temperature rise for FGM if the thickness of each layer is put to be infinitesimal. Second, estimation of a profile function of the thermal diffusivity has been made when inside and rear-surface temperature rises are known. Fair agreement is demonstrated between given and estimated profiles of mixture ratio and thermophysical properties. Finally, estimation of the profile function is considered when only the rear-surface temperature rise is known. An equivalent profile function of the thermophysical properties, which gives the same temperature rise, is obtained although it is quite different from the given profile.

Evaluation of Thermal Barrier Property  
of Functionally Gradient Material  
by Burner Heating Test.

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Department of Materials Processing,  
Faculty of Engineering,  
Tohoku University

**Abstract**

The burner heating test for thermomechanical evaluation of functionally gradient material in a laboratory scale has been described. A temperature difference was given by heating the ceramic side with a burner flame and cooling the metal side with water flow. The damage at the specimen surface was monitored with microscope. A vertical crack formation at the ceramic surface was observed during cooling cycle. The test temperature for the first crack formation was defined as a thermal barrier performance value for the test sample. It has been found that the crack formation temperature is almost constant for the various samples and heating conditions, which indicates a material dependency of the thermal barrier property. The crack formation mechanism was discussed on the basis of a thermal stress analysis using finite element method.

## Evaluation of 8YSZ/NiCrAlY FGM in Arc-Heated Flows

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National Aerospace Laboratory, STA

Masataka YAMAMOTO

National Space Development Agency

### Abstract

The evaluation study of 8YSZ/NiCrAlY FGM materials was carried out in arc-heated high enthalpy flows. Heating rate of  $0.25 \text{ MW/m}^2$  was applied to  $30\phi$  test-pieces which were placed flat to flow direction. Surface and section of exposed test pieces were observed and analyzed by EPMA.

## A Fundamental Evaluation of FGM for High Temperature Rotating Members

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### ABSTRACT

The fundamental evaluation method of the functionally gradient material (FGM) applied to the combined condition of thermal and mechanical load has been developed, which condition is commonly observed in rotating members e.g. gas turbine rotors. The pipe-shaped FGM test pieces were heated from their outer side by an image furnace and simultaneously cooled from their inner side. Under this condition, they were subjected to one-dimensional mechanical loads by a tension test apparatus. Their thermal barrier effect and mechanical damage have been investigated to evaluate the practical property of FGMs. With the combination of 8Y-PSZ and NiCoCrAlY as the two component materials of the FGM, at some hundred  $\text{kW/m}^2$  level heat flux, it has been shown that the FGMs have an equally good bonding property compared with the conventional thermal barrier coating (2 layered TBC) having the same material combination. A fundamental evaluation method under the combined condition has been established.

Evaluation of Cylindrical High Pressure Thrust  
Chambers With Thermal Barrier Coatings

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Fumiei Ono,\* Nobiyuki Yatsuyanagi\* and Hiroshi Takigawa\*

\* Kakuda Research Center, National Aerospace Laboratory, STA

\* Plant & Machinery Div. Nippon Steel Corporation

Abstract

Three cylindrical rocket thrust chambers with conventional ceramics coated layers as thermal barriers were tested using liquid oxygen and gaseous methane propellants at chamber pressures of 3.5 to 5.0 MPa and heat fluxes of 3.5 to 6 MW/m<sup>2</sup>. Two of them were damaged at only one thermal cycle probably due to severe thermal stress. The another one was remained apparently adhered throughout 14 firing tests. In the post-test observation, two spalling modes were inspected. It was thought that nonuniformity of plasma-spray coated layers and severe thermal stress due to the mismatch of top and bond layers caused the spallings.

Functionally Gradient Materials Database

Takayuki Sudo, Katsuto Kisara, Akio Moro, Masayuki Niino  
Kakuda Research Center, National Aerospace Laboratory, STA

Abstract

Functionally gradient materials are promised for space plane. This report mention on the operation and structure of functionally gradient materials database.

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